



09 / 03 903

The
Patent
Office

PCT/GB 00 / 00 5 2 1



INVESTOR IN PEOPLE

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

REC'D 13 APR 2000

WIPO PCT

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

Dated 27 March 2000

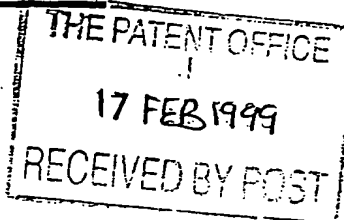
THIS PAGE BLANK (USPTO)

The
Patent
Office

1/77

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference P1351

2. Patent application number 9903474.6
(The Patent Office will fill in this part)

17 FEB 1999

17FEB99 E433805-1 B10007

3. Full name, address and postcode of the or of each applicant (underline all surnames)
UNIVERSITY OF NEWCASTLE
6 Kensington Terrace, Jesmond
NEWCASTLE UPON TYNE

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

75218CC01

4. Title of the invention
PROCESS FOR THE CONVERSION OF A FLUID PHASE
SUBSTRATE BY DYNAMIC HETEROGENOUS CONTACT
WITH AN AGENT

5. Name of your agent (if you have one)
MARKGRAAF PATENTS LIMITED
The Crescent
54 Blossom Street
YORK
YO24 1AP

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Harrison Goddard & Co
Belmont House
20 Wood Lane, Leeds
LS6 2AE
S177 17p/a

Patents ADP number (if you know it)

7499C800C1

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country	Priority application number (if you know it)	Date of filing (day / month / year)
---------	----------------------------------------------	-------------------------------------

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing (day / month / year)
-------------------------------	-------------------------------------

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:
a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

YES

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filling with this form.
Do not count copies of the same document

Continuation sheets of this form

Description

19

Claim(s)

Abstract

Drawing(s)

4

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents
(Please specify)

11.

I/We request the grant of a patent on the basis of this application.

Signature

Philippa M Allen

Date 16.02.1999

MARKGRAAF PATENTS LIMITED

12. Name and daytime telephone number of person to contact in the United Kingdom

Philippa M Allen

01904 610586

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

PROCESS FOR THE CONVERSION OF A FLUID PHASE
SUBSTRATE BY DYNAMIC HETEROGENEOUS CONTACT WITH
AN AGENT

5

The present invention relates to a process for the conversion of a substantially fluid phase substrate by heterogeneous contact with a substantially solid phase agent, the agent for use in the process, an apparatus therefor comprising the agent, novel agent compositions, substrate compositions and the products thereof.

10

In particular the present invention relates to a process for the conversion of a substrate using an agent which is comprised as a rotating surface, the apparatus, agent, and compositions for use therein and the products thereof.

15

The invention makes use of rotating surfaces of revolution technology (hereinafter RSORT) (commonly known as spinning disc technology).

20

The spinning disc concept is an attempt to apply process intensification methods within the fields of heat and mass transfer. The technology operates by the use of high gravity fields created by rotation of a disc surface causing fluid introduced to the disc surface at its axis to flow radially outward under the influence of centripetal acceleration in the form of thin often wavy films. Such thin films have been shown to significantly improve the heat transfer rates. The technology was developed for typical heat and mass transfer operations such as heat exchanging, heating, cooling and mixing, blending and the like, for example as disclosed in R J J Jachuck and C Ramshaw, "Process Intensification: Heat transfer characteristics of tailored rotating surfaces", Heat Recovery Systems & CHP, Vol 14, No. 5, p475-491, 1994.

25

More recently the technology has been adapted for use as a reacting surface for systems which are heat and mass transfer limited, for example for the reaction of substrates which are highly viscous during at least a stage of the reaction and cause problems in achieving good mixing and product yields.

5

Boodhoo, Jachuck & Ramshaw disclose in "Process Intensification: Spinning Disc Polymeriser for the Manufacture of Polystyrene" the use of a spinning disc apparatus in which monomer and initiator is reacted by conventional means to provide a pre-polymer which is then passed across the surface of a spinning disc at elevated temperature providing a conversion product in the form of polymerised styrene.

10

EP 0 499 363 (Tioxide Group Services Limited) discloses another use for spinning disc technology in photo catalytic degradation of organic materials such as hydrocarbons. A solution of salicylic acid and titanium dioxide catalyst was passed across the surface of a rotating disc and irradiated with ultra violet light.

15

These publications therefore disclose the use of spinning disc technology for heating and mass transfer in inert and reactive systems.

20

We have now surprisingly found that spinning disc technology may be further adapted to apply process intensification methods not only within the fields of heat and mass transfer but also within the field of heterogeneous contacting.

25

We have further surprisingly found that the quality of the product obtained is of higher quality than that obtained by conventional processing having, for example, a higher purity or, in polymers, a narrower molecular distribution.

In addition to this, spinning disc technology can be used to obtain products not readily obtainable by other technology.

Accordingly in its broadest aspect there is provided according to the present invention a process for the conversion of a substantially fluid phase substrate by heterogeneous contact of the substrate or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a surface of a support element or part thereof and the support element is adapted to rotate around an axis such that the solid phase agent provides a rotating surface or part thereof and the substrate provides a film flowing substantially radially outward from the axis in dynamic contact with the agent.

Reference herein to a rotating surface is to any continuous or discrete planar or three dimensional surface or assembly which rotates approximately or truly about an axis, and preferably is reference to an approximate or true rotating surface of revolution. An approximate rotating surface of revolution may comprise an asymmetric axis and/or deviation in the surface body and/or circumference creating an axially or radially undulating surface of revolution. A discrete surface may be in the form of a mesh, grid, corrugated surface and the like.

Reference herein to a substantially radially outward flowing film as hereinbefore defined is to any fluid film which may be created by dynamic contact of the fluid phase substrate and the rotating surface as hereinbefore defined, suitably the fluid phase substrate is contacted with the rotating surface at any one or more surface locations and caused to flow outwardly by the action of centrifugal force. A film may be a continuous annulus or may be a non-continuous arc at any radial location. The substrate may provide a

plurality of films in dynamic contact with a rotating surface as hereinbefore defined.

Reference herein to an agent is to any agent which enables, facilitates or
5 otherwise influences conversion of the fluid phase substrate by contact, and
includes a nucleation or growth agent adapted for fluid substrate conversion
by phase change to form crystals or grow seed crystals, a reagent, catalyst or
initiator adapted for fluid phase substrate conversion by reaction to form
products, and the like.

10

The agent may be capable of being immobilised on the surface of a support
element which is adapted to rotate as hereinbefore defined or may be
comprised as an integral part of the surface as part of the construction thereof.
The agent may be adapted to achieve conversion by means of its composition
15 or of its configuration, ie may be a chemical, electronic or like entity or may
be a feature of shape.

Reference herein to a substrate is to the substrate to be converted or its
fragment or derivative formed as part of the contacting with additional
20 substrates, the rotating surface or the agent.

The process of the present invention may be used for any desired
heterogeneous conversion of one or more substrates, or of one or more
fragments or derivatives thereof and is particularly useful for conversions
25 which are heat and/or mass transfer limited. By this means the process of the
present invention is able to substantially improve process performance in
terms of yield and selection of desired products by ensuring that
heterogeneous contact is optimal and occurs at optimum conditions of
temperature, concentration, contact time and the like.

Preferably the process of the invention is a process for conversion of hydrocarbons, such as prepolymerisation, polymerisation, copolymerisation, block polymerisation and the like, upgrading of base hydrocarbons to provide
5 petrochemicals for chemical, fuel or additives applications, for example etherification, dimerisation, alkylation, upgrading low value higher chain (C_{10+}) to high value lower chain branched (C_{2-8}) hydrocarbons, upgrading by decomposition (for example breakdown of salicylic acid) and the like. The process is also admirably suited for adaptation of existing homogeneous
10 processes such as alkylation to form branched hydrocarbons and ethers, to heterogeneous alternatives, using solid acids such as zeolites and the like.

Alternatively the process is a process for the crystallisation of a salt from a solution formed prior to or during contact with the rotating surface.
15

The process may therefore comprise a conversion of one substrate with itself in the presence of an agent as hereinbefore defined or may comprise the conversion of one substrate with one or more additional substrates in the presence of an agent as hereinbefore defined. Substrate(s) is fluid and may be
20 gaseous, vaporous, liquid or fine flowable solid.

The conversion may be carried out entirely or in part under the influence of an added energy source which may be a source of thermal or radiation energy such as ultra violet, micro wave, infra-red, x-ray, ultrasound or at elevated
25 pressure or under the influence of electric or magnetic fields.

The process of the invention as hereinbefore defined may be operated in a single or plural stages. A plural stage conversion may comprise a first pre-conversion stage with further post conversion or upgrading stages, and may be

carried out batchwise with use of a single rotating surface as hereinbefore defined or may be carried out in continuous manner with multiple rotating surfaces in series.

- 5 The process is particularly suited to continuous conversion, and may provide means to perform hitherto batch wise conversions in continuous manner, by means of the improved conversion efficiency and regulated contact time obtained with the invention.
- 10 For example conversions requiring extended contact time may be carried out in continuous manner with use of a recycle of fluid exiting at the periphery of the rotating disc towards the axis of the rotating disc enabling sequential passes of fluid across the surface. In continuous steady state operation an amount of fluid exiting the disc may be drawn off as product and an amount
- 15 may be returned by recycle for further conversion with an amount of fresh substrate feed.

Second or more substrates may be added to the substrate as it passes from one rotating assembly to the next or be added directly to the rotating assembly

20 anywhere between the axis of rotation or the exit from the assembly. In certain cases a multi-step conversion process may be achieved by substrate addition or additions between the axis of rotation and the exit of a single rotating assembly to achieve more than one conversion step in a single pass. It is also possible to have more than one agent on the surface either intermixed or at

25 different radial locations in order to facilitate more than one conversion or conversion stage. These different sections may be controlled at different temperatures and conditions and have different surface geometries as appropriate to the process needs.

Conversion may be carried out with initiation and/or quench as known in the art. It is of a particular advantage of the invention that a plural stage process may employ a pre-mixing stage enabling rapid blending of any diluents or solvent with substrate, or of plural substrates, followed by a conversion stage
5 as hereinbefore defined and a post-conversion quench stage in which reacted product exits the rotating surface at the periphery thereof and is collected in a suitable collection means or on a third stage rotating surface on which quench may be performed by the blending of a quench medium or by cooling. The process of the invention enables rapid homogeneous quenching.

10

It will be apparent that the process of the invention may be controlled both by selection of a specific rotating surface for use as agent support, by nature of immobilisation of agent thereon and by selecting process variables such as
15 temperature, rotation of speed, rate of substrate feed, conversion time and the like. Accordingly the process of the invention provides enhanced flexibility in process control including both conventional control by means of operating conditions, and additionally control by means of rotating surface type and nature of agent immobilisation.

20 Agents may be immobilised on the rotating surface by any means known in the art for supporting agents, suitably by means of spraying such as plasma spraying of solutions of metals, sol gel impregnating with drying and optional calcining or reduction and the like. Agent may be mechanically or chemically immobilised or both. Preferably agent is mechanically coated and chemically
25 immobilised to interact with and bond with the rotating surface in manner to ensure coherent coating with any desired distribution and loading of agent.

Agents may be supported directly on the rotating surface or may be supported on a surface coating of a material adapted to provide surface features or

enhanced surface area. For example the rotating surface may be provided with enhanced surface area in the form of macro surface features such as a corrugated continuous surface for example of a metal foil and the like or micro features such as a coating of microporous material such as zeolite or refractory oxide and the like. The agent may be provided with any additional coating to enhance its conversion performance.

The method of preparing the agent may also be selected according to the nature of the agent itself.

10

Accordingly coating the rotating surface with agent by spraying is particularly suited in case of agents in the form of metal catalyst such as metal or alloy powders which are commonly used for example in hydrogenation reactions and the like.

15

Coating with a sol gel is particularly suited for use with agents in the form of materials which readily form sol gels, such as inorganic oxide catalysts which are commonly used for example in oxidation and partial oxidation reactions.

20 The coating techniques may be used as commonly known in the art, with use of known spray apparatus and dipping methods. In the case of non planar rotating surfaces or rotating surfaces with additional surface features it may be desired to adapt the known techniques to ensure uniformity of coating, and avoid any distortion by virtue of gravitational, meniscal, capillary effects and the like. These effects may suitably be overcome with known methods in which an electrostatic field is applied to one object to be coated during the coating process, or with sol gel techniques in which the colloidal structure of the solution comprising a suspension of solid particles of colloidal dimensions in a liquid is less prone to gravitational effects, meniscal or capillary effects,

optionally additionally mounting the rotating surface in manner to allow vertical rotation during drying and calcining.

5 A RSORT apparatus (commonly known as a spinning disc reactor) generally includes within a conversion chamber a rotating surface or an assembly of a plurality of these which is rotated about an axis to effect transfer of one or more substrates from the axis preferably radially across the rotating surface.

10 An RSORT apparatus as hereinbefore defined comprising a rotating surface as hereinbefore defined has a number of advantageous constructional features according to the present invention.

15 Any suitable feed means may be provided to feed the at least one substrate onto the rotating surface. For example, the feed means may comprise a feed distributor in the form of a "shower head", a "necklace" of outlets for a simple, preferably adjustable, single point introduction such as a "hose-pipe type" feed means. Preferably, the feed means comprises a feed distributor having a plurality of uniformly spaced outlets for the at least one substrate on to the rotating surface as hereinbefore defined.

20

The feed means may be provided at any suitable position with respect to the rotating surface which allows feed of the substrate. For example, the feed means may be axially aligned with the rotating surface for axial feed. Alternatively, the feed means may be positioned such that the feed is spaced
25 from the axis of the rotating surface. Such a position may lead to more turbulence and an enhanced mixing effect. In a further alternative embodiment, feed means may comprise a single feed to a trough which is preferably situated on or co-axial with the axis of rotation of the rotating surface. In this alternative embodiment, substrate flows from the feed outlet

into the trough and is subsequently spread out of the trough on to the rotating surface by centripetal force. In a preferred embodiment, the rotating element as hereinbefore defined comprises a trough situated on the axis of rotation.

5 The trough as hereinbefore defined may be of any suitable shape such as continuous or annular. For example it may have a continuous concave surface comprising part of a sphere, such as a hemispherical surface or it may have an inner surface joined to the rotating surface by at least one connection wall or at least two, in the case where the trough is annular. The inner surface and
10 connection wall may be of any form which allow the function of a trough to be fulfilled. For example the inner surface may be parallel to the rotating surface or concave or convex. The connection wall may comprise a single circular or ovoid wall or a plurality of straight walls. The walls may diverge or converge towards the rotating surface.

15

Preferably, a single circular wall is provided which converges towards the rotating surface to form an undercut trough. This shape generates a reservoir which enhances a circumferential distribution of the substrate flow.

20 Any suitable collection means may be provided for collection of the product as it leaves the rotating surface at its periphery. For example, a receptacle in the form of a bowl or trough at least partially surrounding the rotating element or other fixed part of the apparatus. The collection means may additionally comprise a deflector positioned around the periphery of the rotating surface to
25 deflect product into the collection means. The deflector is preferably positioned at an acute angle to the rotating surface.

The collections means may further comprise outlet means of any suitable form. For example, there may be a single collection trough running around

the periphery of the disc or a collection bowl partially surrounding the rotating element.

5 Outlet means may also be provided in the collection means and these may take the form of apertures of any size and form situated at any suitable position of the collection means to allow egress of the product. In one preferred embodiment, the outlet means are situated to allow vertical egress of the substrate in use.

10 The apparatus may further comprise any suitable control system. Such a control system may regulate the temperature or contact time of substrate and agent by means of speed of rotation, rate of substrate feed and other process parameters to obtain an optimum result.

15 The apparatus as hereinbefore defined may comprise means for optimising conversion conditions. For example, means for imparting an additional movement to the rotating surface, and thus to the substrate, may be provided. Such movement could be in any desired plane or plurality of planes and preferably comprises vibration. Any suitable vibration means may be
20 provided, such as flexible mounting of the surface or off centre mounting, both inducing passive vibration or active vibration means, such as a mechanical element in contact with the rotating element and vibrating in a direction parallel to the rotating element axis. Preferably a passive vibration means is provided in the form of off centre mounting of the rotating element
25 on its axis of rotation. Vibration may alternatively be provided by an ultrasonic emitter in contact with the rotating element for vibration in any desired plane or plurality of planes.

The rotating surface may have any shape and surface formation to optimise conversion conditions. For example the rotating surface may be generally planar or curved, frilled, corrugated or bent. The rotating surface may form a cone or be of generally frusto conical shape.

5

In one preferred embodiment the rotating surface is generally planar and preferably generally circular. The periphery of the rotating surface may form an oval, rectangle or other shape.

- 10 In another preferred embodiment the rotating surface is provided as the inner surface of a cone. The apparatus may comprise at least one cone and at least one other rotating surface or at least one pair of facing cones positioned so as to allow a two stage process of pre-conversion and conversion or pre-blending and conversion with one or more substrates fed to each cone. Preferably
- 15 substrates or pre-reacted substrate exits a smaller cone (or other surface of rotation) in a spray on to the surface of a larger cone (or other surface of rotation) by which it is at least partially surrounded and for the surface of which a further substrate is fed by feed means as hereinbefore defined, to allow mix of the two substrates on the larger rotating surface. Preferably,
- 20 means are provided such that the two cones counter rotate. Such an arrangement enhances mixing and intimate contact of the substrates and reduces the required physical contact time. Alternatively, means are provided such that the cones co-rotate or one is stationary.

- 25 A rotating surface of any shape and surface formation as hereinbefore defined may be provided with surface features which, in addition to the immobilised agent, serve to promote conversion. For example, the surface may be micro or macro profiled, micro or macro porous, non stick, for example may have a release coating, may be continuous or discontinuous and may comprise

elements such as mesh, for example woven mesh, reticulated foam, pellets, cloth, pins or wires, for enhanced surface area, enhanced or reduced friction effect, enhanced or reduced laminar flow, shear mixing of recirculation flow in axial direction and the like.

5

In one preferred embodiment, mixing characteristics of the rotating surface are enhanced by the above features or the like provided on or in the rotating surface. These may be provided in any suitable regular or random arrangement of grids, concentric rings, spider web or like patterns which may be suitable for a given application.

10

Alternatively or additionally to any other surface feature, radially spaced pins in the form of circles or segments of circles may be provided.

15 In another preferred embodiment, a porous surface coating is provided, which aids conversions of certain substrates. Such a coating may be provided in combination with any other of the aforementioned surface features.

20 Surface features in the form of grooves may be concentric or may be of any desired radially spaced form. For example, the grooves may form "wavy" or distorted circles for maximised mixing.

Grooves may be parallel sided, or may have one or both sides which diverge to form undercut grooves or which converge to form tapered grooves.
25 Preferably, the grooves are undercut to promote mixing.

Grooves may be angled to project towards or away from the axis of the rotating surface to enhance or reduce undercut or taper.

Energy transfer means may be provided for the rotating surface or substrate as hereinbefore described. For example heating means may be provided to heat the substrate, for example, as part of the feed means. Additionally, or alternatively heating means may be provided to heat the rotating element in
5 the form of radiant or other heaters positioned on the face of the rotating element which does not comprise the rotating surface for conversion. Preferably, radially spaced, generally circular radiant heaters are provided.

Any preferred cooling or quenching means may be provided in a suitable
10 position to cool the reacted substrate. For example cooling coils or a heat sink may provide cooling by heat exchange, or a reservoir of quench may provide cooling or reaction termination by intimate mixing in the collection means.

15 Cooling means for the substrate may be provided in any suitable position, such as on, in or near the deflector or collection means.

Multiple rotating elements may be provided in an apparatus as hereinbefore defined. For example the rotating elements may be provided in series within
20 the apparatus, with reacted substrate from the first disc being fed directly to at least one further disc. Alternatively or additionally, rotating elements may be provided in parallel with a single reservoir of substrate fed simultaneously to a plurality of discs. In this preferred embodiment a single collection means may be envisaged. The invention is now illustrated in non-limiting manner with
25 reference to the following figures and examples.

Example 1 - Polymerisation of ethylene using a catalyst coated disc.

Phillips catalyst was coated onto the surface of a spinning disc apparatus using methods as described hereinbefore. The coated disc was mounted in a spinning disc apparatus.

The spinning disc apparatus used is shown in diagrammatic form in Figure 1. The main components of interest being:

- 10 i. Top Disc - A smooth brass disc of thickness 17 mm and diameter 500 mm capable of rotating around a vertical axis.
- 15 ii. Liquid Distributor - A circular copper pipe of diameter 100 mm, positioned concentrically over the disc, sprayed fluid vertically onto the disc surface from 50 uniformly spaced holes in the underside. Flowrate was controlled manually by a valve and monitored using a metric 18 size, stainless steel float rotameter. A typical fluid flow rate was 31.3 cc/s
- 20 iii. Motor - A variable speed d.c. motor capable of rotating at 3000 rpm was used. The rotational speed was varied using a digital controller calibrated for disc speeds between 0 and 1 000 rpm. A typical rotational speed was 50rpm.
- 25 iv. Radiant Heaters - 3 radiant heaters (each consisting of two elements) spaced equally below the disc provided heat to the disc. The temperature was varied using a temperature controller for each heater. Each heater temperature could be controlled up to 400°C. Triac

regulators were used to control the speed of the controller response. (These remained on setting 10 throughout the tests).

5 v. Thermocouples and Datascaner - 16 K-type thermocouples embedded in the top disc gave an indication of the surface temperature profile along the disc radius. Odd numbered thermocouples 1 to 15 inclusive were embedded from underneath the disc to a distance 3 mm from the upper disc surface. Even numbered thermocouples, 2 to 16 inclusive were embedded from underneath the disc to a distance 10 mm from the upper disc surface. Each pair of thermocouples i.e. 1 & 2 and 3 & 4 and 5 & 6 etc) were embedded adjacently at radial distances of 85 mm, 95 mm, 110 mm, 128 mm, 150 mm, 175 mm, 205 mm and 245 mm respectively (see Figure 3). The thermocouples were connected to the datascanner which transmitted and logged the data to the PC at set intervals using the DALITE Configuration and Monitoring Software Package.

vi. Manual Thermocouple - A hand-held K-type thermocouple was used to measure the bulk fluid temperature on top of the disc.

20

The rig was used in two arrangements. In one arrangement, feed was constantly added and the heated product was sent to the collection trough. In an alternative arrangement the rig was assembled with a recycle. The fluid flowpath in the recycle arrangement is shown in Figure 4.

25

The spinning disc apparatus of Figure 1 was started up and temperature and rotational speed adjusted. When steady stage was achieved gaseous ethylene was fed to the revolving catalyst coated disc surface at its axis. Product was

withdrawn in the collection trough at the periphery of the disc. Analysis revealed the product was high grade polyethylene.

5 Example 2 – Precipitation of Barium Sulphate Crystals using a disc providing a nucleation/growth rotating surface.

A nucleation surface was provided on the surface of a disc adapted to provide a rotating surface using methods as described hereinbefore. The disc was mounted in a spinning disc apparatus.

10

The spinning disc apparatus used is as described in Example 1. Barium chloride and sodium sulphate solutions were reacted on the surface of the spinning disc. Barium sulphate crystals precipitated out from the solution and the effect of rotational speed on the crystal size and size distribution was studied.

15

Example 3 – Precipitation of Calcium Carbonate Crystals using a disc providing a nucleation/growth rotating surface.

20 A nucleation surface was provided on the surface of a disc adapted to provide a rotating surface using methods as described hereinbefore. The disc was mounted in a spinning disc apparatus.

25 The spinning disc apparatus used is as described in Example 1. Calcium hydroxide and carbon dioxide (gas/liquid) were reacted on the surface of the disc and the effect of rotational speed on the crystal size and size distribution was studied.

Figure 1 illustrates a conventional spinning disc apparatus which is suited for adaptation according to the present invention. The apparatus is enclosed in vessel (1) having at its axis a drive shaft (2) supporting a spinning disc (3). Feed means (4) provides substrate to the surface of the disc (5) at its axis (6).
5 Rotation of the disc causes substrate to flow radially outwards, whereby it contacts the surface of the spinning disc. Fluid is collected at the peripheral edges of the disc by means of collection trough (7) and may be rapidly quenched by means of cooling coils (8). A skirt (9) prevents meniscal draw back of fluid contaminating the drive shaft mechanism. Inlet means (10)
10 enable controlled environment conditions to be provided, for example a nitrogen atmosphere. Outlet vent means (11) enable the venting of atmospheric gases or gases evolved during operation. Observation means are provided by means of windows (12) to observe the progress of the conversion.

15 In Figure 2 is shown a typical spinning disc in the form of a rotating surface according to the present invention comprising an immobilised agent on its surface. The disc (3) is illustrated without any additional surface features and comprises uniform coating (31) which is fused by chemical interaction (32) with the disc surface (33).

20

In Figure 3 is shown an alternative spinning disc in which the agent which it is desired to immobilise is also known as a conventional support material, for example a refractory oxide. In this case the spinning disc (3) is constructed entirely of refractory oxide, the body of which (34) provides a support
25 function and the surface of which (35) provides a catalytic function.

In the process of the invention an apparatus of Figure 1 is started up and operated as described in Example 1. In the case that the process is an exothermic conversion, cooling coils (8) may be used to quench the collected

product in the trough (7). The spinning disc is provided with heating coils (not shown) which may be used to initiate or maintain conversion. The disc or the reactor vessel may be provided with a source of radiation, means for applying an electric or magnetic field and the like as described, at or above the
5 disc surface or at the wall of the reactor vessel.

Figures 5 and 6 show the feed means comprising a trough.

10 In Figure 5 the trough is continuous and forms a well situated on the axis of rotation of the rotating surface. Rotation causes substrate to flow to the wall and form an annular film within the trough.

In Figure 6 the trough is annular and forms a channel co-axial about the axis of rotation of the disc or core. Rotation assisted by the trough profile causes
15 substrate to flow into the trough and to the wall thereof and form an annular film within the trough.

Further advantages of the invention are apparent from the foregoing.

THIS PAGE BLANK (USPTO)

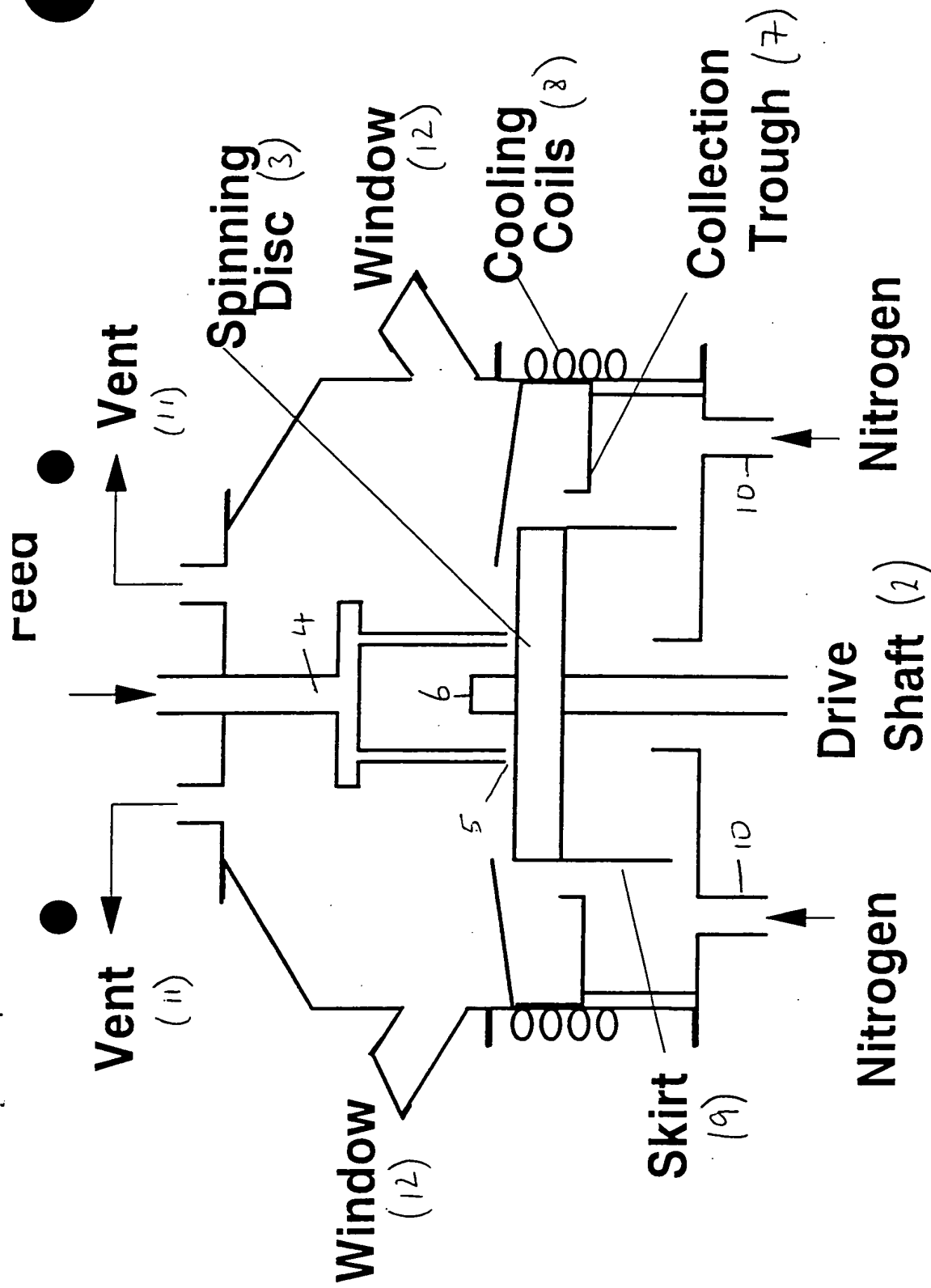


Figure 1 Spinning Disc

THIS PAGE BLANK (USPTO)

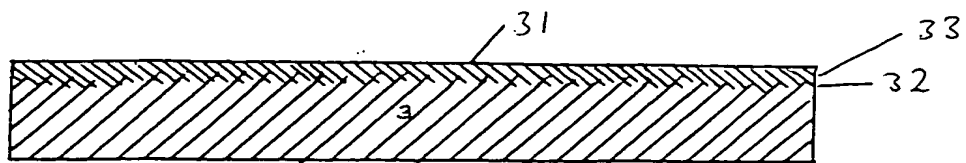


FIGURE 2

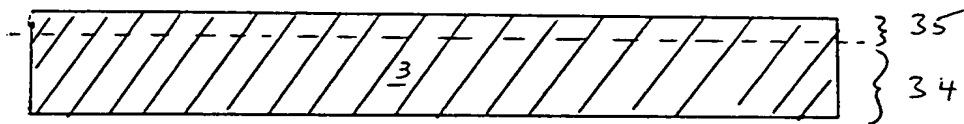


FIGURE 3

THIS PAGE BLANK (USPTO)

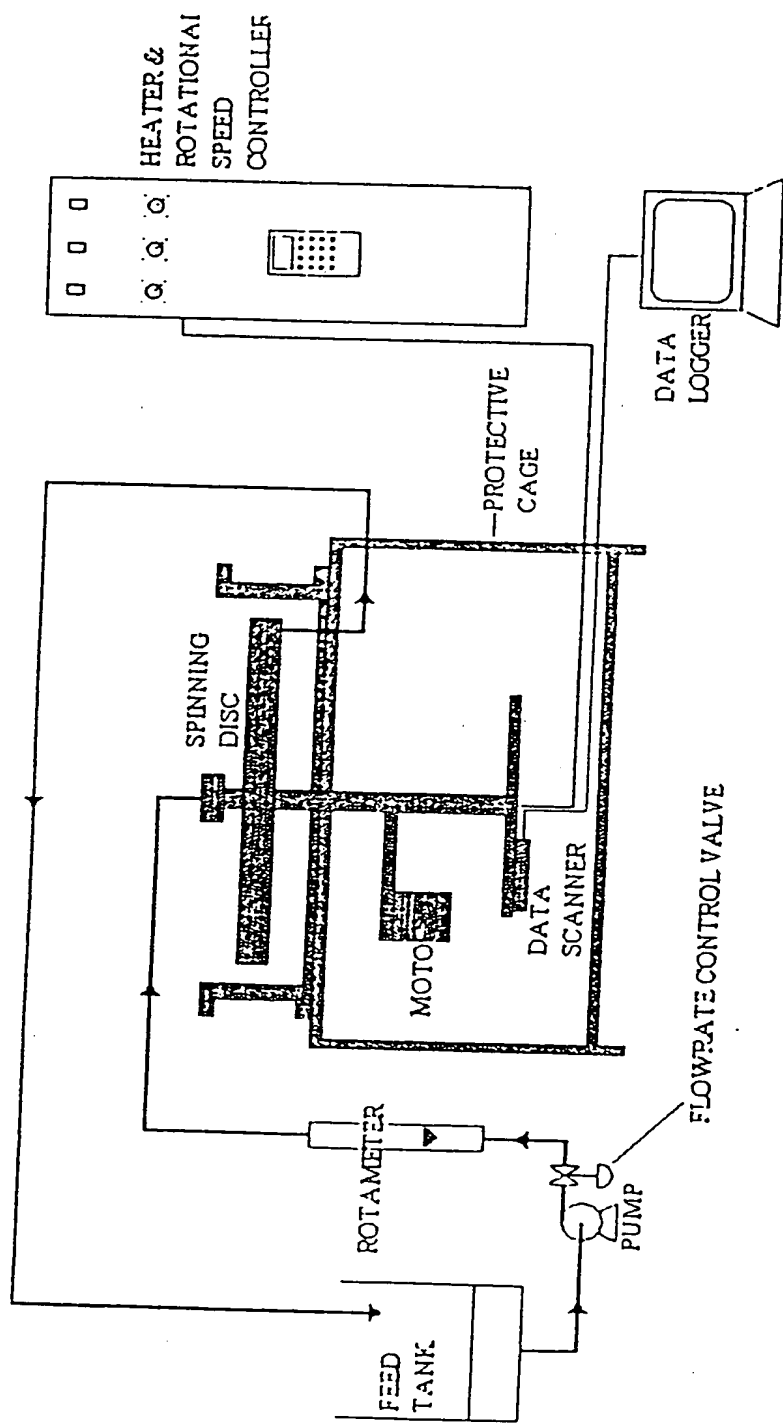


Figure 4 - Diagram of Fluid Flowpath in Recycle Arrangement

THIS PAGE BLANK (USPTO)

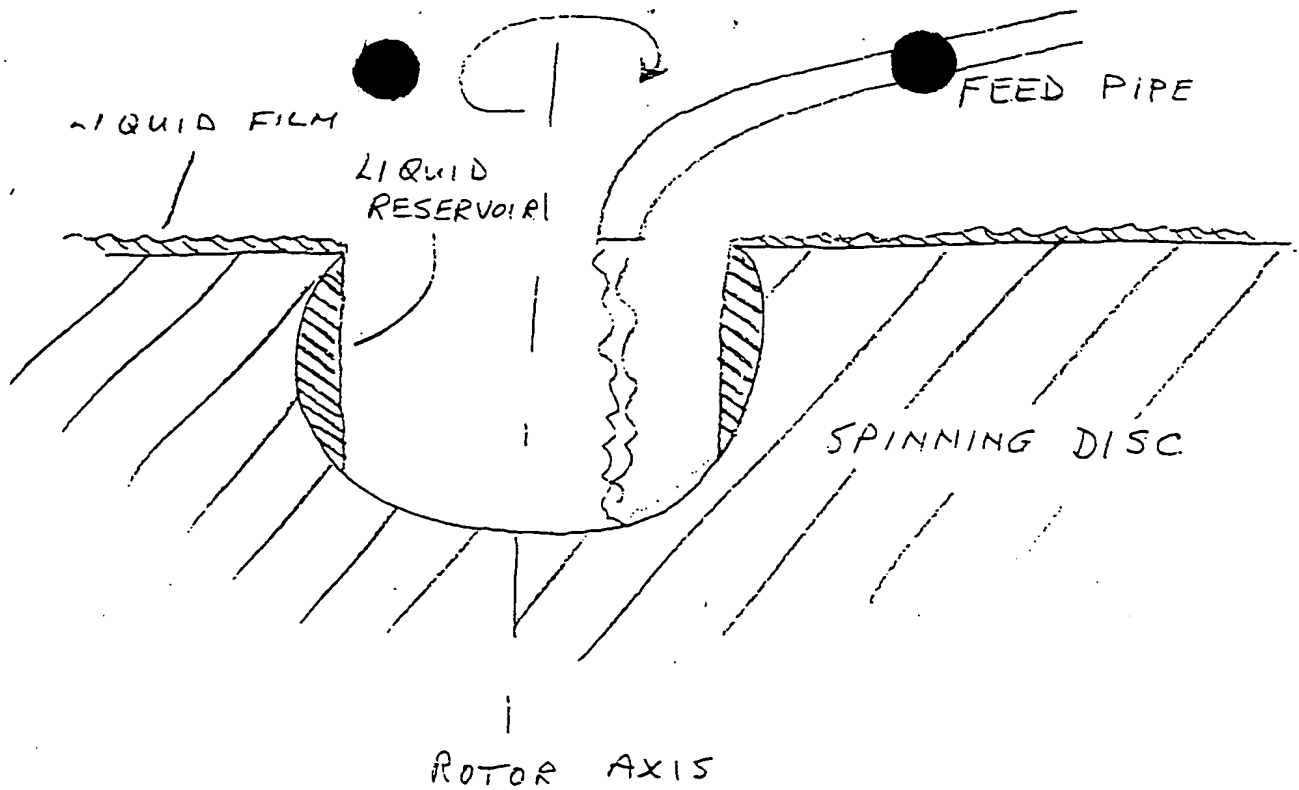


FIGURE 5
CENTRAL SINGLE POINT FEED TO UNDERCUT DIMPLE

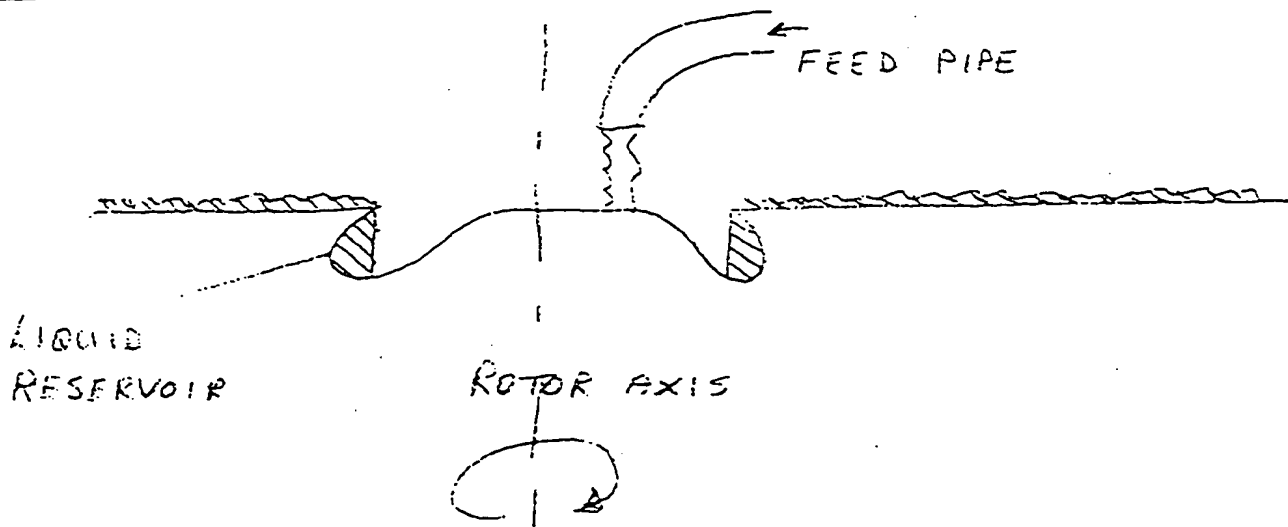


FIGURE 6
SINGLE POINT FEED WITH UNDERCUT CIRCULAR GROOVE

PC7/08001 00521

Harrison-Goddard Foote

17/12/00